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PRELIMINARY SCREENING OF EMULSIFIABLE TOXAPHENE CONCENTRATES[1/]

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In the application of toxaphene emulsions by power spraying, the stability of the emulsion presents only a minor problem. However, where the stock is dipped in a vat the stability of the emulsion is of major importance. Experiments were therefore conducted to develop emulsifiable toxaphene concentrates that would yield stable emulsions. A method for screening candidate concentrates containing different emulsifiers is described, and the results of screening some formulations are reported. Another consideration was the use of agricultural products in insecticide formulations; hence the testing of turpentine and pine oil as solvents.

Formulations Tested

Three types of formulations were tested:

(1) Three experimental concentrates containing 50 percent of toxaphene, 10 percent of emulsifier, and 40 percent of one of the following solvents: kerosene, pine oil (sp. gr. 0.9358 at 15.6°/15.6° C.), or turpentine (all percentages by weight).

(2) Three commercial concentrates:

(A) Toxaphene 65, aliphatic hydrocarbon solvent 25, alkylaryl poplyethylene glycol 7.5, and alkylaryl sulfonate 2.5 percent.

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(B) Toxaphene 60, aliphatic hydrocarbon solvent 22, aromatic hydrocarbon solvent 5, alkylaryl polyethylene glycol 5, polyethylene glycol ester 5, and polyoxyethylene sorbitol oleate-laurate 3 percent.

(C) Toxaphene 50, alkylaryl polyethylene glycol 4, phthalic glycerol alkyd resin 6, epichlorhydrin 0.2, and petroleum distillate 39.8.

(3) A miscellaneous group of formulations suggested by various emulsifier manufacturers.

The specific gravities at 25° C., of the solutions of toxaphene in the experimental concentrates were determined, and for this work were considered as constant with respect to temperature. The volume containing 5 grams of toxaphene for each solution was used for making the test emulsions.

Preparation of Emulsions

The following procedure was adopted for preparing the emulsions:

One gram of emulsifier was weighed into a 50-ml. beaker. The volume of the kerosene, pine oil, or turpentine solution containing 5 grams of toxaphene was pipetted into the beaker and the mixture stirred to dissolve the emulsifier. When necessary, gentle heat was applied. Distilled water was then added slowly until an oil-in-water emulsion was formed. This emulsion was poured into a 500-ml. graduated cylinder and diluted with distilled water to 500 ml. to produce a 1-percent toxaphene emulsion. It was then poured into a 1-liter Erlenmeyer flask, stoppered, and shaken for 30 seconds. Into each of five calibrated vials, 9.7 cm. long and 2.4 cm. inside diameter, 15 ml. of the freshly shaken emulsion was poured. The emulsion was further diluted to contain 0.5 percent of toxaphene by adding to each vial an equal volume of distilled water, tap water (containing 14 grains of calcium carbonate per gallon), 2-percent acetic acid, ammonium hydroxide, or sodium chloride. The vials were placed in racks and left open to the air.

Observations and Screening Criteria

The emulsions were examined after 1 hour, 1 day, and 1 week. Observations were made on the extent of bottom creaming as measured with a millimeter rule, the degree of top clearing, any phase separation, the ease of re-emulsification of the separated oil or ease of resuspension of the cream, and the discernibility of resuspended particles when the vials were illuminated by flashlight.

Since for use in dipping vats a stable emulsion with a slow rate of creaming was desirable, the following criteria were established for choosing the more promising formulations:

(1) The emulsifier was soluble in the toxaphene solution.
(2) No bottom creaming, or only a trace, appeared in any vial at the end of a week.
(3) There was no oil separation in any of the aqueous media, except the 1-percent sodium chloride system, at the end of a week.
(4) The cream was readily redispersed on inversion of the vial.
(5) Upon illumination of the vial after inversion, there were no individually discernible particles.
(6) There was either no top clearing or only slight clearing,

Since no pine-oil concentrates conformed to criterion (2), the pine-oil emulsions chosen for further consideration showed 1 mm. or less of bottom creaming, but met the other criteria.

Results

Experimental concentrates. --In the course of testing the experimental concentrates 103 emulsifiers were tried. The following were found to give satisfactory formulations with at least one type of toxaphene emulsion, according to the criteria outlined above.

Emulsifier	Solvents
Ahco RL 382 (Arnold, Hoffman & Co.)	Kerosene
Ahcowet Anhydrous RS (Arnold, Hoffman & Co.)	Kerosene and turpentine
Antarox D-100 (General Dyestuff Corp.)	Pine oil
Chlorsol (E. F. Drew & Co.)	Pine oil and turpentine
Duponol G (E. I. du Pont de Nemours & Co.)	Kerosene
Emcol H-77 (Emulsol Corp.)	Pine oil
Emulside 75 (Van Dyk & Co.)	Kerosene
Emulsifier 803-M (E. F. Drew & Co.)	Turpentine
Emulsifier R (Monsanto Chemical Co.)	Kerosene
Nopco Agrimul 60 (Nopco Chemical Co.)	Kerosene and turpentine
Nopco 1219-A (Nopco Chemical Co.)	Kerosene
Nopco HBX-2 (Nopco Chemical Co.)	Kerosene
Skil 181-A (Gallowhur Chemical Corp.)	Kerosene
Tenlo 400 (Griffin Chemical Co.)	Kerosene and turpentine
Trex 45 (Griffin Chemical Co.)	Turpentine
Trex 80 (Griffin Chemical Co.)	Turpentine

The kerosene-type concentrates containing Emulsifier R, Tenlo 400, or Duponol G were stable in 1-percent sodium chloride solution for a week. Many of the concentrates that did not meet these criteria may be suitable for other purposes, such as use in sprays.

Commercial concentrates. --The amounts of bottom creaming in emulsions prepared from the three commercial concentrates after they had stood for 7 days are shown below.

Diluent	A	B	C
Distilled water	Trace	1 mm.	1 mm.
Sodium chloride	Broken	Trace	Broken
Acetic acid	Broken	2 mm.	2 mm.
Ammonium hydroxide	2 mm	1 mm.	3 mm.
Tap water	1 mm.	Trace	3 mm.

Emulsions A and C showed nearly complete top clearing and particles were discernible upon resuspension.

It is apparent that concentrates A and C do not meet the criteria established. Except for extent of creaming, concentrate B does meet these criteria.

This report does not constitute a condemnation of any of these products. Two of them are being used extensively and successfully in Texas, although there have been reports (Radeleff and Bushland 1950) of cattle killed by dipping in vats in which such emulsions had deteriorated.

Miscellaneous formulations. --Of 23 miscellaneous formulations tested, 19 were either recommended by emulsifier producers or supplied by them for evaluation; the remaining 4 were arbitrarily compounded in the laboratory. Formulations containing the following emulsifiers and solvents were found to be satisfactory.

Emulsifier	Percent	Solvent	Percent
Emcol H-66	35	Deodorized kerosene	15
Antarox A-200	50	None	
Antarox B-290	50	None	
Antarox B-201	35	Butyl Cellosolve	15
Ahcowet RS	50	None	
	60	None	
Ahco RL-382	16	Kerosene	26
G-8910 T (Atlas Powder Co.)	50	None	
	45	Kerosene	10

All formulations except those containing Antarox A-200 or Ahco RL-382 formed solubilized systems when diluted with water. The degree of transparency varied, but the most opaque materials showed no sign of creaming at the end of a week in the vials. Formulations containing Ahcowet RS and Ahco RL-382 broke in 1-percent sodium chloride solution.

Experiments in miniature vats.--The validity of this method for selecting formulations for further development has been supported by two experiments conducted in miniature (7-gallon) concrete dipping vats. In the first experiment[3/] four formulations prepared in the laboratory and two commercial concentrates (A and C) were used. Under similar conditions of vat-charging methods, climate, contamination, and time, the laboratory-prepared formulations showed less deterioration than the commercial formulations.

Two of the miscellaneous solubilizable concentrates were included in these tests. Although they showed no oil separation or gumminess, one of them developed a thick pellicle at its surface. The other concentrate showed evidence of creaming, but upon agitation the cream was resolubilized.

Conclusions

These experiments indicate that certain combinations of emulsifier, solvent, and toxaphene produce emulsifiable concentrates that are more stable than some commercial formulations when diluted and held under laboratory conditions. It is to be emphasized that most of the results were obtained in laboratory glassware. The confirmatory tests with emulsions held in miniature vats do not represent actual field observations. However, the formulations that performed better under those limited experimental conditions merit further testing in ranch vats.

Literature Cited

Radeleff, R. D., and Bushland, R. C.
1950. Acute toxicity of chlorinated insecticides applied to livestock. Jour. Econ. Ent. 43(3): 358-364.

3/ Conducted by R. D. Radeleff (unpublished report).